QUALITY ASSURANCE PROJECT PLAN FOR GROUNDWATER MONITORING

Liberty/Bosma and Cow Palace Dairy Facilities, Granger, Washington

Prepared for:

The Law Offices of Charles M. Tebbutt, P.C.

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Appendices

List of Acronyms

Acronym	Description
AOC	Administrative Order on Consent
DQO	Data Quality Objective
EPA	United States Environmental Protection Agency
QA	quality assurance
QC	quality control
QAPP	Quality Assurance Project Plan
SOG	Standard Operating Guideline
WET	Water and Environmental Technologies, PC

1.0 PROJECT MANAGEMENT

Water and Environmental Technologies, PC (WET) has prepared this Quality Assurance Project Plan (QAPP) on behalf of the Law Offices of Charles M. Tebbutt, P.C. for the collection and analyses of groundwater samples from monitoring wells in and around the Liberty/Bosma and Cow Palace Dairy Facilities (Facilities). The Facilities are located in the Yakima River Valley near Granger, Washington.

The activities covered under this QAPP include the collection and analysis of groundwater samples from any wells installed by either the United States Environmental Protection Agency (EPA) or the Yakima Valley Dairies (Dairies) to satisfy requirements of Administrative Order on Consent (AOC) SDWA-10-2013-0080. Wells installed by EPA are referred to herein as EPA wells, and wells installed by the Dairies are referred to as AOC wells.

1.1 PROJECT/TASK ORGANIZATION

This section identifies the individuals participating in the project and discusses their specific roles and responsibilities.

The Project Manager is responsible for scheduling field activities, directing the activities of the project team, and overall implementation of this QAPP. The Project Manager is the principal decision maker for the project.

The Project Engineer will either conduct or oversee all groundwater monitoring on EPA and/or AOC wells. The Project Engineer is a licensed Professional Environmental Engineer in the state of Washington and will be the principal data collector for the project.

Laboratory analysis of groundwater samples collected from EPA and/or AOC wells will be performed by Cascade Analytical, Inc. of Union Gap, Washington (Cascade Analytical). Cascade Analytical is accredited by the Washington Department of Ecology.

The Quality Assurance (QA) Manager will review and validate all data collected from EPA and/or AOC wells. Quality assurance/quality control (QA/QC) procedures are discussed in Section 2.5, and data validation procedures are discussed in in Section 4.0.

1.2 PROBLEM DEFINITION/BACKGROUND

1.2.1 Purpose

This QAPP was developed to document the planning, implementation, and assessment procedures of the project, and how specific QA and QC activities will be applied during the project.

1.2.2 Objectives

The primary objective of this sample effort is to determine concentrations of nitrates and other constituents in groundwater samples collected from EPA and AOC wells.

1.3 PROJECT DESCRIPTION AND SCHEDULE

The project includes monitoring EPA and AOC wells. Monitoring will include measuring static water levels and water quality parameters and collecting groundwater samples for laboratory analysis.

1.3.1 Project Description

The proposed sample effort includes the following tasks:

- Manually gauge the static water level in EPA and AOC wells using a water level meter;
- Measure water quality parameters in groundwater from EPA and AOC wells;
- Collect groundwater samples from EPA and AOC wells; and
- Analyze groundwater samples for the constituents listed in Section 2.4 at a laboratory certified in Washington State.

1.4 QUALITY OBJECTIVES AND CRITERIA

1.4.1 Project Quality Objectives

The data generated during this sample effort will be used primarily to determine the concentrations of the constituents listed in Section 2.4 at each EPA/AOC well location.

1.4.2 Measurement Performance Criteria

Data generated during this sample effort will be evaluated for precision, accuracy, and completeness during the data review, validation, and verification process. These activities are discussed in detail in Section 4.0.

1.5 SPECIAL TRAINING/CERTIFICATION

Field personnel will be trained according to WET's Corporate Health and Safety Plan and will conduct the sample effort in accordance with the Site Specific Health and Safety Plan. The Site-Specific Health and Safety Plan will identify the potential hazards that may be encountered during the project, list techniques to communicate hazards to management and other field personnel, and prescribe controls to mitigate hazards.

Cascade Analytical will analyze groundwater samples collected during this effort. Cascade is accredited by the Washington Department of Ecology for the analytical parameters that will be measured during the project. A certificate of accreditation and analytical method list is provided in Appendix A.

1.6 DOCUMENTS AND RECORDS

Field daily logs, field logbooks, or other field forms (e.g., groundwater purge and sample forms, boring/well logs, etc.) will be maintained to document the collection of every sample. At the time of sampling, the appropriate sample container will be selected, and the sample number for each sample will be recorded on the field form by the sampler. Any QC samples collected will also be noted on the daily field log, in the field logbook, or on other field forms, as appropriate.

At the end of each day, and prior to the transfer of the samples offsite, chain-of-custody forms will be completed. The chain-of-custody form will indicate the sample identification, sample type, date and time of collection, the specific analyses requested for that sample, and the signature of the sample collector. When more than one form is needed, the forms will be sequentially numbered.

Samples will be transmitted to the laboratory with completed chain-of-custody forms. Copies of the forms will be retained by the sampler and transferred to the Project Manager for filing. Original chain-of-custody forms will remain with the samples during storage and analysis, and signed originals will be forwarded with the data packages to Water & Environmental Technologies.

2.0 DATA GENERATION AND ACQUISITION

2.1 SAMPLING DESIGN

The primary objective of the project, determining groundwater concentrations, will be achieved by collecting and analyzing groundwater samples from EPA and AOC wells. Sampling methods are discussed below.

2.2 SAMPLING METHODS

2.2.1 Static Water Level Measurement

Static water levels will be manually gauged in each well using a water level meter, and each measurement will be recorded in a field log book or appropriate field form.

2.2.2 Groundwater Sample Collection

Groundwater samples will be collected from monitoring wells using the low-flow purge and sample procedures presented in SOG-8 in Appendix B. During purging, water quality parameters (temperature, pH, specific conductivity, oxidation-reduction potential, and dissolved oxygen) will be measured as described in SOG-5 Appendix B.

2.2.3 Equipment Decontamination

Sampling equipment will be decontaminated as described in SOG-2 in Appendix B.

2.3 SAMPLE HANDLING AND CUSTUDY

2.3.1 Groundwater Sample Labeling

Sample labels will be filled out with waterproof ink at the time of sample collection. Sample containers will be labeled with the following information:

- Sampler (Water & Environmental Technologies)
- Project name
- Sample identification
- Sample depth (if appropriate)
- Preservative
- Sampling date and time

Identification numbers for groundwater samples will correspond to the well name. QC samples will be labeled similarly to natural samples so they are indistinguishable to laboratory personnel.

2.3.2 Chain of Custody Procedures

The key aspect of documenting sample custody is recordkeeping. Field daily logs, field logbooks, or other field forms (e.g., groundwater purge and sample forms, boring/well logs, etc.) will be maintained to document the collection of every sample. At the time of sampling, the appropriate sample container will be selected, and the sample number for each sample will be recorded on the field form by the sampler. Any QC samples collected at this time will also be noted on the daily field log, in the field logbook, or on other field forms, as appropriate.

Sample containers will be placed on ice in a cooler for temporary sample storage. At the end of each day, and prior to the transfer of the samples offsite, chain-of-custody forms will be completed. The chain-of-custody form will indicate the sample identification, sample type, date and time of collection, the specific analyses requested for that sample, and the signature of the sample collector. When more than one form is needed, the forms will be sequentially numbered.

Samples will be transmitted to the laboratory with completed chain-of-custody forms. Copies of the forms will be retained by the sampler and transferred to the Project Manager for filing. Original chain-of-custody forms will remain with the samples during storage and analysis, and signed originals will be forwarded with the data packages to Water & Environmental Technologies.

2.3.3 Sample Packaging, Handling, and Shipment

Prior to shipment, sample containers will be securely packed inside plastic coolers. Each sample container will be wrapped with bubble wrap or Styrofoam packing and placed on absorbent pads (if water is present in samples) or other suitable packing material that has been placed in the bottom of the cooler. Ice will be placed in the cooler to keep samples cold, if required. Packing material will be added to fill the cooler completely and secure sample containers in an upright position.

The original chain-of-custody form(s) will be enclosed in plastic and placed inside the cooler. Samples will be packaged and either hand-delivered or shipped to the appropriate analytical laboratories according to U.S. Department of Transportation (DOT) and commercial carrier regulations. Additional information regarding sample packaging and shipping is provided in SOG-3 in Appendix B.

2.4 ANALYTICAL METHODS

Natural groundwater samples will be collected in laboratory-supplied containers, submitted to Cascade Analytical, Inc. in Union Gap, Washington, and analyzed for any or all of the following constituents and methods:

Constituent	**Method**
Chloride	EPA300.0_2.1_1993
Nitrate	EPA300.0_2.1_1993
Nitrite	EPA300.0_2.1_1993
Sulfate	EPA300.0_2.1_1993

Constituent	**Method**
Orthophosphate	EPA300.0_2.1_1993
Total Kjeldahl Nitrogen	A4500 N org
Total Phosphorus	EPA365.1
Ammonia	EPA350.1
Alkalinity	SM2320 B-97
Calcium	EPA200.7
Magnesium	EPA200.7
Sodium	EPA200.7
Potassium	EPA200.7

QA/QC samples will be submitted to the same laboratory as their associated natural samples and analyzed for the same constituents as the natural samples.

In addition to laboratory analyses, selected samples may be screened in the field for nitrate and/or ammonia concentration using test strips or a Hach® Pocket Colorimeter™ II and compound-specific reagent packets (Method 8039 – Cadmium Reduction). All field screening supplies and equipment will be operated, calibrated, and maintained according to manufacturer recommendations, and the Cadmium Reduction Method 8039 is provided in Appendix B.

2.5 QUALITY CONTROL

2.5.1 QC Sample Collection

Quality Control samples will be collected to assess field and laboratory operations and to evaluate overall precision and accuracy throughout the project. Field QC samples will include field duplicate and equipment rinsate blank samples and will be collected at a frequency of one duplicate and one blank per 20 natural samples. Duplicate samples will be collected by splitting a natural sample in the field, and equipment rinsate blanks will be collected by pouring laboratory supplied deionized water through or over sampling equipment and into a laboratory-supplied container.

2.5.2 Laboratory Quality Control

Laboratory QC procedures generally include those identified in the analytical methods, as well as analyzing blank and duplicate samples, laboratory control samples (LCS), and matrix spike (MS) samples to evaluate the accuracy and precision of the analytical results. For LCS and MS samples, the sample is prepared by adding known amounts of target constituents to a natural parent sample and analyzing it. Accuracy is measured by comparing the known amounts with the measured amounts of these constituents. Precision, or reproducibility, is evaluated by comparing laboratory control and matrix spike sample recoveries with laboratory control sample duplicates (LCSD) and matrix spike duplicates (MSD) sample recoveries.

2.5.3 Instrument/Equipment Testing, Inspection, and Maintenance

Sampling instruments and equipment are identified in the SOG's in Appendix B. Instruments and equipment are inspected prior to each use and maintained on an as-needed basis.

2.5.4 Instrument/Equipment Calibration and Frequency

Sampling instruments and equipment are calibrated according to manufacturer's recommendations and as described in the applicable SOGs in Appendix B. The dates, times, and values associated with instrument calibration will be recorded in field log books or the appropriate field forms.

2.5.5 Inspection/Acceptance of supplies and Consumables

Sampling supplies and consumables primarily consist of sample containers provided by the laboratory. Sample containers will be new and clean. All supplies and consumables will be visually inspected by either the Project Manager or the Project Engineer prior to their use. The supplies and consumables will be determined to be acceptable for use if they appear intact, clean, and free of defect.

2.5.6 Non-direct Measurements

The only outside data sources that will be utilized for implementation of this QAPP is a Groundwater Monitoring Well Installation Report prepared by the Dairies containing the locations and construction details of EPA and AOC wells. Applicable data from this report includes, but is not limited to, well identification numbers, surveyed well locations and top-of-casing elevations, and reported static water levels.

2.5.7 Data Management

Data collected during the sample effort will be recorded in field log books or the appropriate field forms, and analytical data will be delivered by the laboratory in hard copy or pdf format, or both. All project data will be transferred to electronic, modifiable format after completion of field activities and laboratory analyses.

3.0 ASSESSMENT AND OVERSIGHT

3.1 ASSESSMENTS AND RESPONSE ACTIONS

Assessments to be used during the project include performance evaluations and data quality assessments. Performance evaluations of field personnel will be conducted by the Project Manager throughout sampling activities via direct observation. Acceptable performance will be determined if field personnel adhere to this QAPP, the Site Specific Health and Safety Plan, and applicable industry standards. Results of performance evaluations will be provided verbally to field personnel after each evaluation. If performance of field personnel is determined to be unacceptable, the Project Manager has the authority to stop work immediately until necessary corrective actions are implemented. Corrective actions will be verified by repeated performance evaluations.

Data quality assessment procedures are described in Section 4.0.

3.2 REPORTS TO MANAGEMENT

Daily tailgate meetings or teleconferences will be held at the beginning of each work day to communicate to the Project Manager the status of the sample effort, including the number of natural and QC samples collected to date, number of laboratory shipments, and any quality assurance or health and safety-related concerns. Discussion topics of daily meetings will be recorded in field log books.

4.0 DATA VALIDATION AND USABILITY

This section discusses the QA activities that will occur after data collection is completed. Implementation of these actions is conducted to verify that the data conform to the specified criteria, thus achieving the project objectives.

4.1 DATA REVIEW

Field and laboratory data generated during this sample effort will be reviewed, verified, and validated by the QA Manager. Field data entered into databases will be verified. Errors identified during the verification of data will be corrected prior to release of the final data.

The laboratories are responsible for verifying analytical results prior to the submittal of the final laboratory data report to WET. Initially, all analytical data generated by the laboratories are verified by the laboratories. During the analysis process, the analyst and the laboratory QA Manager verify that the results have met various performance-based control limits (e.g., surrogate recoveries and continuing calibration). Non-conformance of various method QC requirements and control limits warrants the re analysis and/or re-extraction of a sample.

Once the laboratory has released the final data report, the data will be reviewed by WET personnel to verify that all samples are accounted for. Finally the data will be verified and validated based on the quality objectives specified in applicable portions of EPA's Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review (EPA 1999; 2002). If data do not meet required criteria, they will be flagged with data qualifiers as specified under the action portion of each requirement of the functional guidelines (EPA 1999; 2002).

For analytical data for which no validation guidelines exist (e.g., natural attenuation parameters), the same objectives, criteria, evaluating procedures, and actions will be used.

4.2 DATA VERIFICATION AND VALIDATION

During field activities, the Project Engineer will be responsible for overseeing field measurements and data recording. The laboratory will forward chain-of-custody forms to WET personnel upon receipt of samples. The WET data reviewer will review the chain of custody forms to verify correct analyses are being performed. In addition, the data reviewer will verify that samples were received at the laboratory at the appropriate temperature and in good condition (e.g., no excessive headspace, broken sample containers, etc.). If a sample does not arrive at the laboratory at the appropriate temperature or the integrity of the sample is in question, the potential implication of the anomaly will be evaluated and a course of action will be determined. The condition of the samples at the time of sample receipt will be noted in the QA Report discussed below.

In addition, data verification and validation will be conducted to assess the laboratory's performance in meeting the quality objectives and performance based criteria specified in the analytical methods. Data validation will be conducted in accordance with applicable sections of EPA's Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review (EPA 1999; 2002). Data validation procedures will entail evaluating the following components:

- Holding times (verify that samples were analyzed within the specified holding time).
- Laboratory method blank samples [verify that no analytes were present in method blank samples and that a blank was analyzed every 20 samples (or more often) for each matrix].
- Field blank samples (verify that no analytes were present in the field blank samples).
- Matrix spikes/matrix spike duplicate samples (verify that matrix spike and matrix spike duplicates were analyzed every 20 samples for each matrix or at least for each batch of samples, where applicable, and that control limits were met).
- Surrogate percent recoveries (very that surrogate recoveries met control limits).
- Laboratory check samples (verify that laboratory check sample results, if submitted, met control limits).
- Laboratory duplicate samples (verify that duplicate analyses were conducted every 20 samples for each matrix or at least for each batch of samples, where applicable, and that control limits were met).
- Field duplicate samples (calculate relative percent differences for each set of field duplicate samples).

If data do not meet the quality criteria, they will be flagged with data qualifiers as specified under the action portion of each requirement of the functional guidelines (EPA 1999; 2002). Typical data qualifiers include, but are not limited to, "J," used to indicate an estimated value, "B," used to indicate blank contamination, and "R," used to indicate a rejected value. Upon completion of the data validation, a QA report will be prepared summarizing the findings and addressing whether the requirements for each analysis have been met. Limitations to the usability of the data will also be discussed in the report. The QA report will be summarized in the applicable project deliverable(s).

Data qualifiers that have been assigned to the data will be entered into the project databases. These entries will also be verified by a second individual.

4.3 RECONCILIATION WITH USER REQUIREMENTS

The goal of data validation is to ascertain the quality of each data point and to identify data points that do not meet data quality criteria identified in the functional guidelines. During data validation, data that do not meet the quality objectives and criteria may be qualified as estimated or rejected. Rejected data will not be used for any purpose. An explanation of any rejected data will be included in the project reports.

Data qualified as estimated will be used for all intended purposes. These data will be presented in the databases with the appropriate data qualifier(s). These data may be less precise or less accurate than unqualified data. The data users will evaluate the effect of the inaccuracy or imprecision of the qualified data in assessing the overall project.

5.0 REFERENCES

U.S. Environmental Protection Agency. 1983. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020. U.S. EPA, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency. 1999. Contract Laboratory Program National Functional Guidelines for Organic Data Review. EPA 540/R-99/008. U.S. EPA, Office of Emergency and Remedial Response, Washington, DC.

U.S. Environmental Protection Agency. 2002. Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. EPA 540-R-01-008. U.S. EPA, Office of Emergency and Remedial Response, Washington, DC.

APPENDIX A

CASCADE ANALYTICAL, INC. CERTIFICATE OF ACCREDATION

The State of Washington Department of Ecology

Cascade Analytical Inc. - Yakima
Union Gap, WA

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an ACCREDITED LABORATORY for the analytical parameters listed on the accompanying Scope of Accreditation. This certificate is effective May 29, 2013 and shall expire May 28, 2014.

Witnessed under my hand on May 29, 2013

Alan D. Rue
Lab Accreditation Unit Supervisor

Laboratory ID
C858

WASHINGTON STATE DEPARTMENT OF ECOLOGY

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

SCOPE OF ACCREDITATION

Cascade Analytical Inc. - Yakima

Union Gap, WA

is accredited for the analytes listed below using the methods indicated. Full accreditation is granted unless stated otherwise in a note. Accreditation for U.S. Environmental Protection Agency (EPA) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" (SW-846) is for the latest version of the method. SM refers to EPA approved editions of “Standard Methods for the Examination of Water and Wastewater.” ASTM is the American Society for Testing and Materials. Other references are described in notes.

Matrix/Analyte	Method	Notes
Drinking Water		
Bromide	EPA 300.0_2.1_1993	
Chloride	EPA 300.0_2.1_1993	
Fluoride	EPA 300.0_2.1_1993	
Nitrate	EPA 300.0_2.1_1993	
Nitrate + Nitrite	EPA 300.0_2.1_1993	
Nitrite	EPA 300.0_2.1_1993	
Orthophosphate	EPA 300.0_2.1_1993	
Sulfate	EPA 300.0_2.1_1993	
Color	SM 2120 B-01	
Turbidity	SM 2130 B-01	
Alkalinity	SM 2320 B-97	
Hardness, Total (as CaCO3)	SM 2340 C-97	
Specific Conductance	SM 2510 B-97	
Solids, Total Dissolved	SM 2540 C-97	
Total Organic Carbon	SM 5310 C-00	1
Fecal coliform-count	SM 9222 D (m-FC)-97	
Total Coli/Ecoli - count	SM 9223 B (Colilert® QTray)	
Total Coli/Ecoli - detect	SM 9223 B Colilert	
Non-Potable Water		
Solids, Total Volatile	EPA 160.4_1971	
Bromide	EPA 300.0_2.1_1993	

Matrix/Analyte	Method	Notes
Chloride	EPA 300.0_2.1_1993	
Fluoride	EPA 300.0_2.1_1993	
Nitrate	EPA 300.0_2.1_1993	
Nitrate + Nitrite	EPA 300.0_2.1_1993	
Nitrite	EPA 300.0_2.1_1993	
Orthophosphate	EPA 300.0_2.1_1993	
Sulfate	EPA 300.0_2.1_1993	
Color	SM 2120 B-01	
Turbidity	SM 2130 B-01	
Alkalinity	SM 2320 B-97	
Hardness, Total (as CaCO3)	SM 2340 C-97	
Specific Conductance	SM 2510 B-97	
Solids, Total Dissolved	SM 2540 C-97	
Solids, Total Suspended	SM 2540 D-97	
Biochemical Oxygen Demand (BOD)	SM 5210 B-01	
Total Organic Carbon	SM 5310 C-00	1
Fungicides	CAI 9061	2
Fecal coliform-count	SM 9222 D (m-FC)-97	
Total Coli/Ecoli - count	SM 9223 B (Colilert® QTray)	

Accredited Parameter Note Detail

(1) Interim accreditation pending the successful completion of an on-site audit to verify method capabilities (WAC 173-50-100). (2) Provisional accreditation pending on-site assessment and PT completion.

05/20/2013

Authentication Signature
Alan D. Rue, Lab Accreditation Unit Supervisor

Date

APPENDIX B

STANDARD OPERATING GUIDELINES AND FIELD SCREEN METHODS

STANDARD OPERATING GUIDELINE

SOG-2: EQUIPMENT DECONTAMINATION

INTRODUCTION

This guideline describes field procedures typically followed to decontaminate sampling and monitoring equipment. Proper decontamination procedures minimize the potential for cross-contamination among sampling points.

EQUIPMENT

- Two or three containers (e.g., 5-gallon buckets, or 5- or 10-gallon plastic tubs) for dip rinsing, washing, and collection of rinse water.
- Two or three utility brushes or test tube brushes to remove visible contamination. A test tube brush (or similar) can be stapled to the end of a dowel and used to clean the inside of a bailer.
- Non-phosphate detergent to mix with potable or distilled water.
- Deionized, distilled, and/or potable water. Note: Rinse solutions other than water may be required depending on the type of contaminant or contaminant concentration encountered. For example, a methanol rinse solution may be required to decontaminate equipment that has been in contact with petroleum [i.e., light non-aqueous phase liquid (LNAPL)] or other recalcitrant substances (i.e., oils/tars) that cannot be adequately removed using a non-phosphate detergent. Refer to task-specific work plans for use of other solvents, if any, which may be used for equipment decontamination.
- Multi-gallon storage containers filled with potable water for rinsing or washing.
- Spray bottles, squirt bottles, or garden sprayers to apply rinse liquid. Use a separate bottle for each liquid.
- Protective gloves.
- Paper towels to wipe off gross contamination.
- Garbage bags, other plastic bags, or aluminum foil to wrap clean sampling equipment after decontamination, store sampling equipment, or dispose of decontamination debris.
- Sample bottles for rinsate blanks.
- Department of Transportation (DOT)-approved container (e.g., 55-gallon drum) to store decontamination wash and rinse water.
- Steam cleaner with power source and water supply.

PROCEDURES

In most cases, the following procedures are adequate to remove contamination.

1. Pre-clean sampling equipment. If there is gross contamination on equipment, wipe it off with paper towels and/or rinse it off with water. Additional internal decontamination may be possible by circulating water or cleaning solutions inside the equipment.

2. Wash individual parts of equipment with detergent water and scrub with brushes. When appropriate, take equipment apart to remove visible contamination.

3. Double-rinse the sampling equipment with distilled water. If needed, apply a rinse solution of methanol and a final rinse with distilled or deionized water.

4. Wipe sampling equipment with a paper towel or allow it to air dry.

5. Place small sampling equipment in clean plastic bags or sealed containers, or wrap the equipment in aluminum foil for storage.

Note: For heavy equipment, brushes and/or a steam cleaner may be required in some cases to decontaminate sampling equipment. Steam cleaners are most often used for decontaminating large equipment such as downhole drill rig equipment (e.g., augers). Brushes may also be used to remove dry residual soil from drill rigs and excavator buckets.

EQUIPMENT (RINSATE) BLANKS

As specified in the Facility-Wide Sampling and Analysis Plan (SAP) and Quality Assurance Project Plan (QAPP), equipment (rinsate) blank samples will be prepared for submittal to the analytical laboratory. Equipment blank samples should be collected at a frequency specified in the Facility-Wide SAP/QAPP. The procedure for preparing equipment (rinsate) samples is as follows:

1. Pour laboratory-provided analyte-free water through or over the sampling equipment.

2. Collect the rinsate water in a clean bottle.

3. Pour the collected rinsate water into the appropriate sample container(s).

4. Instruct the laboratory to conduct the same analyses on the rinsate blank samples as the other samples being analyzed.

SPECIAL NOTES

To reduce the potential for cross-contamination, samples should be collected so that the least contaminated location is sampled first. Subsequent sampling should be completed in the order of increasing contamination. Areas that typically contain lower levels of contamination include those upgradient of the source, background areas, and the periphery of the contaminated area.

Monitoring instruments that are exposed to sampled materials must also be decontaminated. They should be washed or at least rinsed before monitoring other sampling locations.

STANDARD OPERATING GUIDELINE
SOG-3: SAMPLE PACKAGING AND SHIPPING
(SOIL, SEDIMENT, AND WATER)

INTRODUCTION

This guideline presents methods for shipping non-hazardous materials, including most environmental soil, sediment, and water samples, via United Parcel Service (UPS), Federal Express, and Greyhound. Many local laboratories offer courier service as well.

EQUIPMENT

- Coolers or ice chests
- Sorbent material
- Bubble wrap
- Strapping tape
- Labels and pens
- Chain-of-custody forms
- Chain-of-custody seals
- UPS, Federal Express, or Greyhound shipping forms.

Samples shipped to the analytical laboratory can be sent via UPS or Federal Express on a next-day basis. Greyhound bus service should be used only if direct service is available.

PROCEDURES

1. Place absorbent pads in the bottom of the shipping container to absorb liquids in case of sample container breakage. Use one absorbent pad for each quart of liquid that is being shipped.
2. Wrap glass jars or bottles in plastic bubble wrap.
3. Leave a small amount of air space in any plastic sample container to prevent the cap from coming off if the container is compressed.
4. Pack volatile organics analysis (VOA) vials in protective wrap or foam holders.
5. Choose a method of sample chilling that will not physically or chemically damage the collected samples. Re-usable blue ice blocks, block ice, or ice cubes are acceptable methods. Use sufficient chilling material to keep the samples cool from the time of sample collection, throughout the sampling activities, and for the duration of the shipment (up to 48 hours) to the analytical laboratory. Samples should be chilled to 4 degree Celsius (ºC) $\pm$ 2 ºC.
6. Use waterproof pens and labels to identify the sample containers, and cover each label with clear tape after identification.
7. Band the cooler closed with strong adhesive tape and apply custody seals.

8. Cover the drain plug with adhesive tape to prevent any liquid from escaping.
9. Prior to shipping the samples, review the chain-of-custody form and bottle labels to check that the required entries are filled. Sign and date the chain-of-custody form and enter the time the samples are released to the shipping agency or analytical laboratory. When more than one chain-of-custody form is needed, they will be sequentially numbered.
10. If multiple coolers are needed for shipping a single batch of samples, one chain-of-custody form can be placed inside one cooler. However, a label should be attached to each cooler in the batch indicating the total number of shipping containers and which container contains the original chain-of-custody form.
11. Throughout the sampling activities and during transport to the analytical laboratory, the samples will be maintained in the secure custody of the sampling team, shipping agency, and/or analytical laboratory personnel. The sampling team will retain copies of all chain-of-custody forms. Original chain-of-custody forms will remain with the sample during storage, shipping, and analysis and will be forwarded with the final analytical reports to Water & Environmental Technologies personnel.
12. Original chain-of-custody forms will be enclosed in plastic and taped to the inside of the cooler lid.

SPECIAL NOTES

Specific requirements for packaging may apply if the samples are known to be hazardous materials as defined in 49 CFR 171.8 (samples are not considered hazardous waste; therefore, manifest requirements do not apply). Identify on sample label and/or chain-of-custody form if any of the samples are known or suspected to contain hazardous or listed wastes.

Samples retained but not chosen for analysis by the analytical laboratory may be assessed a disposal fee. A disposal fee is frequently assigned to samples, typically soil, that has been retained beyond standard analytical holding periods. Therefore, consult with the Project Manager to determine which samples may be of interest, and contact the selected analytical laboratory regarding its disposal policies. Arrangements may be made with the analytical laboratory to return unanalyzed samples for later disposal to the area of origin. Samples containing hazardous or listed wastes will be appropriately disposed of by the analytical laboratory and/or owner in accordance with state and federal laws.

If samples are shipped via UPS (or similar service), UPS will not be required to sign the chain-of-custody form. The chain-of-custody form signed by field personnel will already be sealed inside the cooler and secured with a custody seal.

Custody seals will not be used if the samples are picked up from the sampling location by the analytical laboratory, or a sampling technician delivers the samples directly to the analytical laboratory.

STANDARD OPERATING GUIDELINE

SOG-5: MEASUREMENT OF FIELD PARAMETERS: pH, DISSOLVED OXYGEN, SPECIFIC CONDUCTANCE, TURBIDITY OXIDATION-REDUCTION POTENTIAL, AND TEMPERATURE

INTRODUCTION

This guideline describes the procedures typically used by Water & Environmental Technologies, PC personnel to measure the pH, dissolved oxygen, specific conductance, turbidity oxidation-reduction potential (ORP, also referred to as redox potential), and temperature of ground- or surface water.

Equipment

- Multi-parameter water quality meter
- Flow-through cell or plastic cup
- Transport/calibration cup
- Probe sensor guard
- Operations manual
- Spare batteries
- Standard conductivity calibration solutions [447, 1413, 2074, 8974 microSiemens per centimeter (µS/cm)]
- pH buffers (4.00, 7.00, 10.00)
- ORP calibration solution
- Pens, field logbook, and/or appropriate field forms (e.g., groundwater purge and sample form)

PROCEDURES

Calibrate multi-parameter water quality meter at the office prior to commencement of field activities to check instrument is in proper working order. At a minimum, calibrate before use each day (or more frequently as necessary) as indicated below. The initial daily calibration may be performed at the office (if located in proximity to the site), motel, or in the field.

1. Press the On/Off Key. Check the battery charge indicator located at the bottom of the liquid crystal display (LCD) screen. Replace batteries if the battery charge indicator is low.
2. Calibrate the meter according to the manufacturer's instructions. Note: The meter must be calibrated for each field parameter in accordance with the instructions in the operations manual at the beginning of each sampling day. Additional calibrations may be performed during the day if deemed necessary.
3. Connect the probe sensor to the flow-through cell. If the flow cell is not used, make sure the probe sensor guard is installed.

4. Begin passing water into the flow-through cell. If the flow-through cell is not used, place the probe module into a sample of the water or directly into the body of water being evaluated. Be sure to completely immerse all sensors into the water.
5. Provide a constant flow of fresh water across the probe module to actuate readings.
6. Observe the meter's LCD display, and record the values on the groundwater purge and sample form or field logbook.
7. Remove the probe from the water and rinse (soak) with distilled water.
8. Place the probe sensor in the transport/calibration cup with 1/2 inch of potable water for short-term storage. The transport/calibration cup should be sealed to prevent evaporation.

STANDARD OPERATING GUIDELINE

SOG-8: Groundwater Sampling

INTRODUCTION

This guideline provides the procedures typically followed by Water & Environmental Technologies personnel during groundwater sampling of monitoring wells.

EQUIPMENT

- Electric water level monitoring probe.
- Multi-phase interface monitoring probe.
- Bladder pump, peristaltic pump, pre-cleaned, disposable, 2- or 4-inch bailers with disposable cord, inertial pump, submersible pump, or other suitable sampling device.
- Flexible tubing [polyethylene (PE), Teflon™, or similar]
- Multi-parameter water quality meter.
- Nitrocellulose filters (if conducting field filtering).
- Sample containers (laboratory-supplied) with appropriate preservatives.
- Additional chemical preservatives (if necessary).
- Watch or stopwatch.
- Sample labels, pens, field logbook, or other appropriate field forms (e.g., groundwater purge and sample forms, chain-of-custody forms), and access agreements and third-party sample receipts (if warranted).
- Monitoring well construction data (for well screen intervals).
- Sample shipping and packaging supplies (refer to SOG-3).

PROCEDURES

Record the data and information collected during this procedure on an appropriate groundwater purge and sample form.

1. Perform the following prior to groundwater sampling:
    a. Calibrate the water-quality meter, prior to beginning sampling and as necessary based on field conditions, in accordance with the instructions in the manufacturer's operation manual.
    b. Examine the monitoring well to be sampled for any structural damage, poorly fitting caps, and leaks into the inner casing.
    c. Record an initial measurement of the depth to water. Static water levels should be measured and recorded no more than 24 hours prior to the sampling event. Calculate the volume of water in the well casing if volume-based purging is to be used. Total well depth should not be measured at the start of a sampling event (due to the potential to cause turbidity). Measure total well depth after sample collection.

d. If light non-aqueous phase liquid (LNAPL) is potentially present, measure the depth and thickness of the LNAPL and the static water level. Calculate the corrected groundwater level if LNAPL is present.

2. Use one of the following devices for purging:

   a. Dedicated bladder pump: place the pump intake at a depth approximately equal to the middle or just slightly below the middle of the well screen interval or water column unless another position is justified based on site-specific conditions.

   b. Peristaltic pump: place the pump intake at a depth equal to the approximate middle or just slightly above the middle of the well screen interval or water column unless another position is justified based on site-specific conditions. Note: If degassing of water is occurring when sampling with a peristaltic pump, alternative types of sampling equipment will be used for volatile organic compound (VOC) or volatile petroleum hydrocarbon (VPH) sample collection.

   c. Inertial pump: place the pump intake at a depth approximate to the middle or just slightly below the middle of the well screen interval or water column unless another position is justified based on site-specific conditions. Note: Inertial pumps shall not be used for collecting samples for VOC and VPH analyses.

   d. Submersible pump: place the pump intake at a depth approximate to the middle or just slightly below the middle of the well screen interval unless another position is justified based on site-specific conditions.

   e. Pre-cleaned or disposable bailers.

   f. Another suitable purging/sampling device.

3. Purge the well using one of the following methods. If a well exhibits evidence of slow recharge, or produces excessively silty water, etc., the well will be redeveloped at least 5 days prior to a sampling event.

   a. Well-volume-based purging and sampling.

      (1) Establish a purging rate to pump or bail approximately three well-casing volumes of groundwater without dewatering the well.

      (2) If using a pump, set-up the discharge tubing, flow-through cell, water quality meter, and purge water collection container. A purge water, activated carbon filtration system may also be used and will be placed inline and downstream of the flow-through cell. If using a bailer, maintain a clean plastic container next to the well for collecting observation samples. Begin purging the well.

      (3) At the beginning of purging and periodically thereafter, record the following water quality parameters/observations on the groundwater purge and sample form:

         - Date and time
         - Purge volume and/or flow rate
         - Water depth
         - Temperature
         - pH
         - Specific conductance
         - Dissolved oxygen

- Oxidation-reduction potential (ORP)
- Turbidity
- Other observations as appropriate (color, presence of odors, sheen, etc).

(4) Continue purging until three or more successive measurements of water quality parameters have stabilized and a minimum of three well-casing volumes of water have been evacuated from the well. See note at end of this SOG regarding well stabilization criteria.

(5) Collect the sample.

(6) If sampling using a bailer, allow a 4- to 6-inch column of water to be purged from the bottom of the bailer before filling the appropriate sample containers. If the collected water is very turbid, or a bottom-emptying bailer is not used, properly transfer the water from the bailer into the appropriate sample containers. Be careful to avoid agitating the sample. When sampling for volatile organic compounds (VOCs), turn the bottle upside down to identify possible headspace. If bubbles are present, resample.

b. Low-flow purging and sampling.

(1) Place the pump intake at a depth equal to the approximate middle or just slightly above the middle of the well screen interval or water column.

(2) Place an electronic water level indicator probe in the well, approximately 0.5 to 3 inches below the piezometric surface.

(3) Connect the pump discharge tube to a flow-through cell housing a water quality parameter probe.

(4) Activate the pump for purging at a flow rate ranging from approximately 0.1 to 0.5 liters per minute (L/min), as measured by timing the rate at which the flow-through cell is filled.

(5) During purging, monitor the water level in the well to evaluate potential drawdown. The minimal drawdown goal is less than approximately 4 inches. If drawdown is observed (especially rapid drawdown at the beginning of purging), decrease the pumping rate.

(6) Measure water quality parameters (including, temperature, pH, specific conductance, turbidity, ORP, and dissolved oxygen) at approximately 3 to 5-minute intervals during purging. Continue purging until three or more successive measurements of water quality parameters have stabilized. See note at end of this SOG regarding well stabilization criteria. If these measurements do not indicate stabilization, continue purging for approximately 30 minutes.

(7) Immediately after purging, collect the sample using the same flow rate that was used during purging unless it is necessary to decrease the rate to minimize aeration or turbulent filling of sample bottles.

4. If the well contains measurable LNAPL, make sure the pump intake is placed in the upper 2 feet of water column and collect the samples without purging in a manner that discourages mixing of the groundwater sample with air or LNAPL. If a groundwater sampling is required from wells containing LNAPL for the purposes of characterizing chlorinated VOCs in the alluvial aquifer, purge the well prior to sampling unless or until LNAPL becomes entrained in the sampling apparatus. If LNAPL will likely become entrained in the groundwater, the

sample should be collected without purging. If LNAPL becomes entrained in the sampling apparatus then the sampling effort for VOCs can be aborted.

5. Samples collected for field analysis using a Hach colorimeter or samples collected for dissolved metals will be filtered using a 0.45 µm filter prior to analysis.
6. When multiple analyses will be performed, collect the samples in order of decreasing sensitivity to volatilization (e.g., VOC samples first and metals last). When sampling VOCs, turn the bottle upside down to identify possible headspace. If bubbles are present, resample. If possible, the pump should not be moved or turned off between purging and sampling; however, the pump may need to be turned off for a very brief period (as a practical matter) so field personnel can handle samples and minimize the potential for water to splash on the ground surface.
7. If a well purges dry, let it recover to 80 percent of original water column, then sample. If the well takes a very long time to recover (i.e., longer than 2 hours), try to sample the well at the end of day or first thing the next day. If the well has not recovered to the point that by the following day there is sufficient water to collect a sample, the well will be considered "dry" and no sample will be collected.
8. Label and fill the appropriate containers according to the anticipated analyses.
9. Follow the sampling packaging and shipping procedures outlined in SOG-3.
10. Follow personnel and equipment decontamination procedures outlined in SOG-1 and SOG-2.

WELL STABILIZATION CRITERIA

Wells will be considered stable when the criteria listed in the following table have been met for pH, specific conductance, and temperature. Attempts will also be made to stabilize ORP and dissolved oxygen.

Field Parameters	Stabilization Criteria for Three or More Consecutive Readings	Notes
pH	Difference between three or more consecutive readings is within ±10%	–
Temperature	Difference between three or more consecutive readings is within ±10%	–
Specific Conductance	Difference between three or more consecutive readings is within ±10%	–
ORP	Difference between three or more consecutive readings is within ±10%	Very sensitive. Attempts will be made to achieve stabilization; however, due to parameter sensitivity this may not be possible.

Dissolved Oxygen	Difference between three or more consecutive readings is within ±10% or within 10% of 0.2 milligrams per liter (mg/L), whichever is greater	Very sensitive. Attempts will be made to achieve stabilization, especially when collecting samples of VOC analysis; however, due to parameter sensitivity this may not be possible.

Attempts will be made to achieve the stabilization criteria. If field parameter measurements do not indicate stabilization, continue conventional purging until a minimum of three well-casing volumes have been evacuated or continue low-flow purging for approximately 30 minutes.

QUALITY CONTROL

When sampling groundwater, do not purge between the collection of original samples and the collection of duplicate samples. Original and duplicate samples are collected sequentially, without appreciable delay between collection cycles.

WELL ENCLOSURE SEAL

During groundwater monitoring events, rubber seals in the well enclosures shall be observed for signs of wear and leakage. Rubber seals that do not appear to be providing an appropriate seal will be replaced according to the manufacturer specifications.

Nitrate

DOC316.53.01066

Cadmium Reduction Method **Method 8039**

0.3 to 30.0 mg/L NO_3^-N (HR) **Powder Pillows or AccuVac® Ampuls**

Scope and application: For water, wastewater and seawater.

Test preparation

Instrument-specific information

The tables in this section show all of the instruments that have the program for this test. Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests.

To use either table, select an instrument, then read across to find the corresponding information for this test.

Table 1 Instrument-specific information for powder pillows

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

For best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to get the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.

This method is technique-sensitive. Shaking time and technique influence the color development. For most accurate results, use a standard solution that is within the test range and run the test several times. Increase or decrease the shaking time to get the expected result. Use the adjusted shaking time for sample measurements.
The reagents that are used in this test contain cadmium. Rinse the sample cell immediately after use to remove all cadmium particles. Collect the reacted samples for proper disposal.
A deposit of unoxidized metal will remain at the bottom of the sample cell after the reagent dissolves. The deposit will not affect results.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used and use any recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Use the Safety Data Sheets for disposal information for unused reagents. Consult the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Powder pillows

Description	Quantity
NitraVer® 5 Nitrate Reagent Powder Pillow, 10-mL	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 8 for reorder information.

AccuVac Ampuls

Description	Quantity
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

Refer to Consumables and replacement items on page 8 for reorder information.

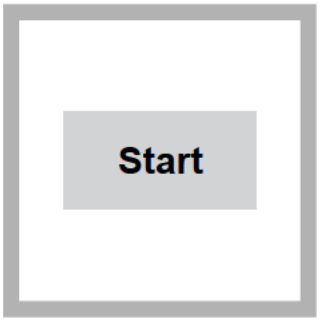
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If prompt analysis is not possible, immediately filter and keep the samples at or below 6 °C (43 °F) for a maximum of 48 hours.
- To preserve samples for up to 28 days, adjust the sample pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and keep at or below 6 °C (43 °F). The test results then include nitrate and nitrite.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5.0 N sodium hydroxide standard solution.
- Correct the test result for the dilution from the volume additions.

Powder pillow procedure

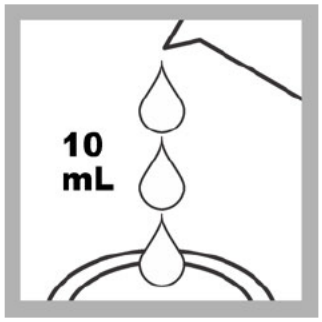
⚠CAUTION

Hazardous waste exposure. Prepared samples contain cadmium. Refer to the SDS for safe handling and disposal instructions. Obey all local and regional disposal regulations.



1. Start program **355 N, Nitrate HR PP**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

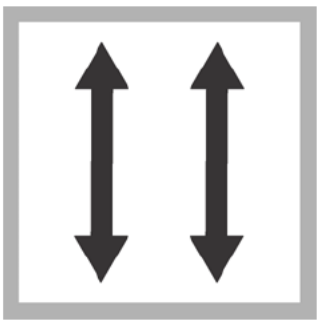
Note: *Although the program name may vary between instruments, the program number does not change.*



2. Prepare the sample: Fill a sample cell with 10 mL of sample.

3. Add .

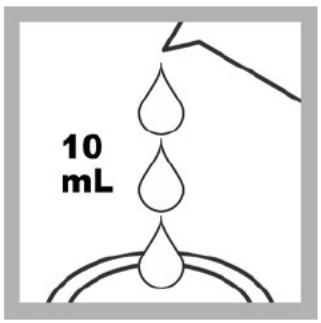
4. Start the instrument timer. A 1-minute reaction time starts.



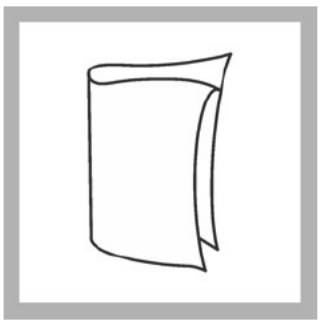
5. Close the sample cell. Shake the cell vigorously until the timer expires. Some powder may not dissolve. Undissolved powder will not affect results.

6. Start the instrument timer. A 5-minute reaction time starts.

An amber color shows if nitrate is present.

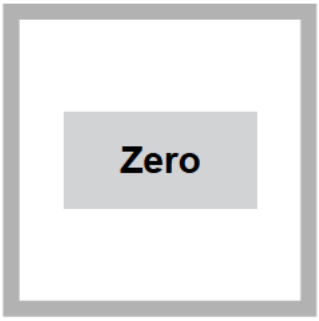


7. Prepare the blank: When the second timer expires, fill a second sample cell with 10 mL of sample.

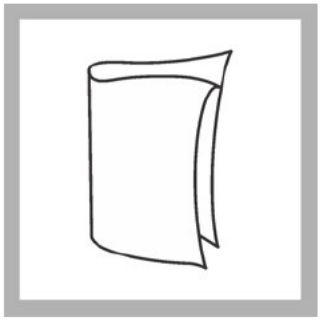


8. Clean the blank.

9. Insert the blank into the cell holder.

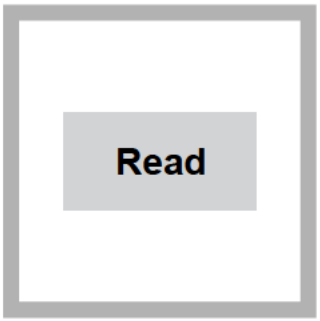


10. Push **ZERO**. The display shows 0.0 mg/L NO_3^-–N.



11. Clean the prepared sample.

12. Within one minute after the timer expires, insert the prepared sample into the cell holder.

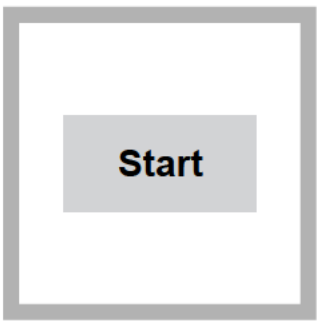


13. Push **READ**. Results show in mg/L NO_3^-–N.

AccuVac Ampul procedure

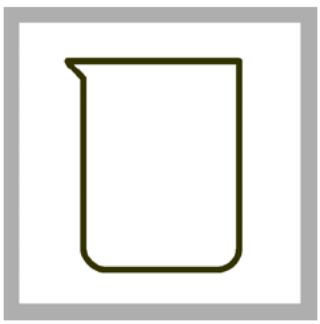
⚠CAUTION

Hazardous waste exposure. Prepared samples contain cadmium. Refer to the SDS for safe handling and disposal instructions. Obey all local and regional disposal regulations.



1. Start program **361 N, Nitrate HR AV**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

Note: *Although the program name may vary between instruments, the program number does not change.*



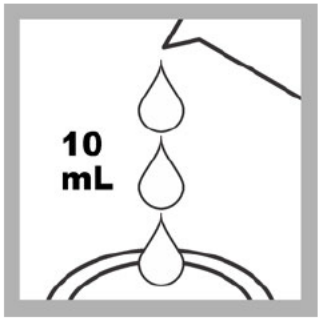
2. Prepare the sample: Collect at least 40 mL of sample in a 50-mL beaker.

3. Tap the bottom of a NitraVer 5 Nitrate AccuVac Ampul to dislodge the powder. Fill the Ampul with sample. Keep the tip immersed while the Ampul fills completely.

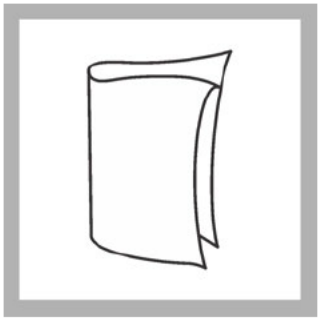
4. Start the instrument timer. A 1-minute reaction time starts.

5. Invert the Ampul 48 to 52 times as the timer counts down.

6. Start the instrument timer. A 5-minute reaction time starts.
Keep the sample still while the timer counts down. An amber color shows if nitrate is present.

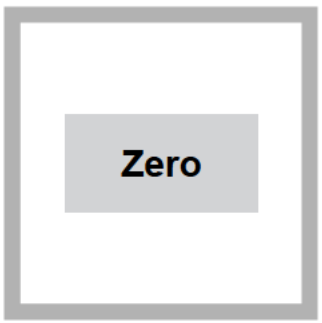


7. Prepare the blank: When the second timer expires, fill a sample cell with 10 mL of sample.

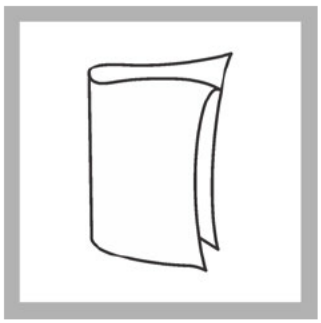


8. Clean the blank.

9. Insert the blank into the cell holder.

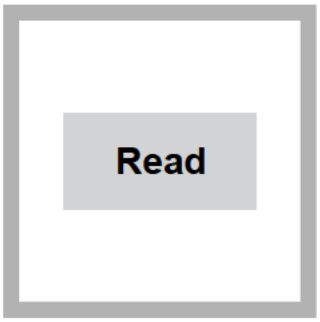


10. Push **ZERO**. The display shows 0.0 mg/L NO_3^-–N.



11. Clean the AccuVac Ampul.

12. Within one minute after the timer expires, insert the prepared sample AccuVac Ampul into the cell holder.



13. Push **READ**. Results show in mg/L NO_3^-–N.

Interferences

Interfering substance	Interference level
Chloride	Chloride concentrations above 100 mg/L cause low results. The test can be used at high chloride concentrations (seawater) if a calibration is made with standards that have the same chloride concentration as the samples (refer to Seawater calibration on page 6).
Ferric iron	Interferes at all levels

Interfering substance	Interference level
Nitrite	Interferes at all levels Compensate for nitrite interference as follows: **1.** Add 30-g/L Bromine Water by drops to the sample until a yellow color remains. **2.** Add one drop of 30-g/L Phenol Solution to remove the color. **3.** Use the test procedure to measure the concentration of the treated sample. Report the results as total nitrate and nitrite.
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pretreatment may be necessary.
Strong oxidizing and reducing substances	Interfere at all levels

Seawater calibration

Chloride concentrations above 100 mg/L cause low results. To use this method for samples with high chloride concentrations, calibrate the instrument with nitrate standard solutions that contain the same amount of chloride as the samples.
Prepare calibration standards that contain chloride and 1.0, 3.0, 5.0 and 10.0 mg/L nitrate (as NO_3^-–N) as follows:

1. Prepare 1 liter of chloride water that has the same chloride concentration as the samples.
   a. Weigh the applicable amount of ACS-grade sodium chloride: (chloride concentration of samples in g/L) x (1.6485) = g of NaCl per liter.
      Note: *18.8 g/L is the typical chloride concentration of seawater.*
   b. Add the sodium chloride to a 1-liter volumetric flask.
   c. Dilute to the mark with deionized water. Mix thoroughly. Use this water as the dilution water to prepare the nitrate standard solutions.
2. Use a pipet to add 1.0, 3.0, 5.0 and 10.0 mL of a 100 mg/L nitrate-nitrogen (NO_3^-–N) standard solution into four different 100-mL Class A volumetric flasks.
3. Dilute to the mark with the prepared chloride water. Mix thoroughly.
4. Complete the test procedure for each of the standard solutions and for the prepared chloride water (for a 0-mg/L standard solution).
5. Measure the absorbance of the standard solutions and enter a user calibration into the instrument.
6. Use the user program to measure samples that contain high concentrations of chloride.

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- 1000 mg/L Nitrate Nitrogen (NO_3^-–N) Standard Solution
- 100-mL volumetric flask, Class A
- 25-mL volumetric pipet, Class A and pipet filler
- Deionized water
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Prepare a 250 mg/L nitrate-nitrogen standard solution as follows:
   a. Use a pipet to add 25 mL of a 1000 mg/L nitrate nitrogen standard solution into a 100-mL volumetric flask.

b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.

2. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
3. Go to the Standard Additions option in the instrument menu.
4. Select the values for standard concentration, sample volume and spike volumes.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the prepared standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.

   Note: *For AccuVac® Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of the prepared standard solution to three 50-mL portions of fresh sample.*
6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Standard solution method

Use the standard solution method to validate the test procedure, reagents and instrument.

Items to collect:

- Nitrate Nitrogen Standard, Solution, 10.0-mg/L NO_3^-–N

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users may get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
355	10 mg/L NO_3^-–N	9.3–10.7 mg/L NO_3^-–N	0.3 mg/L at 0 ppm, 0.5 mg/L at 10 ppm, 0.8 mg/L at 30 ppm NO_3^-–N
361	10 mg/L NO_3^-–N	9.3–10.7 mg/L NO_3^-–N	0.5 mg/L at 0 ppm, 0.6 mg/L at 10 ppm, 0.8 mg/L at 30 ppm NO_3^-–N

Summary of method

Cadmium metal reduces nitrate in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt. The salt couples with gentisic acid to form an amber colored solution. The measurement wavelength is 500 nm for spectrophotometers or 520 nm for colorimeters.

Pollution prevention and waste management

Reacted samples contain cadmium and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
NitraVer® 5 Nitrate Reagent Powder Pillows, 10-mL	1	100/pkg	2106169
OR			
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1	25/pkg	2511025

Required apparatus (powder pillows)

Description	Quantity/test	Unit	Item no.
Stopper, Neoprene, solid, size #1	2	12/pkg	1480801
OR			
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Required apparatus (AccuVac)

Description	Quantity/test	Unit	Item no.
Beaker, 50-mL	1	each	50041H

Recommended standards

Description	Unit	Item no.
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3--N	500 mL	30749
Nitrate Nitrogen Standard Solution 1000 mg/L NO_3--N	500 mL	1279249
Wastewater Influent Standard, Mixed Parameter, for NH_3-N, NO_3-N, PO_4, COD, SO_4, TOC	500 mL	2833149
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
Bromine Water, 30 g/L	29 mL	221120
Cylinder, mixing, 50-mL	each	2088641
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet, Volumetric, Class A, 0.5 mL	each	1451534
Pipet, volumetric, Class A, 1.00-mL	each	1451535
Pipet, volumetric, Class A, 2-mL	each	1451536
Pipet, volumetric, Class A, 3-mL	each	1451503
Pipet tips for TenSette Pipet 1970001	50/pkg	2185696
Pipet tips for TenSette Pipet 1970001	1000/pkg	2185628
Phenol Solution, 30-g/L	29 mL	211220
Pipet, volumetric, Class A, 25-mL	each	1451540
Pipet filler, safety bulb	each	1465100
AccuVac® Snapper	each	2405200
Sodium Hydroxide, 5 N	50 mL	245026

Consumables and replacement items (continued)

Description	Unit	Item no.
Sulfuric Acid, ACS	500 mL	97949
Flask, volumetric, Class A, 100-mL	each	1457442

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